

**REDUCTION OF HYDROGEN CYANIDE  
CONCENTRATIONS AND ACUTE  
INHALATION TOXICITY FROM  
FLEXIBLE POLYURETHANE FOAM  
COMBUSTION PRODUCTS BY THE  
ADDITION OF COPPER COMPOUNDS**

**PART III. THE EFFECT OF COPPER  
ADDITIVES ON THE FLAMMABILITY  
CHARACTERISTICS OF FLEXIBLE  
POLYURETHANE FOAM**

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**U.S. DEPARTMENT OF COMMERCE  
National Institute of Standards  
and Technology  
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**Sponsored by:  
International Copper Association, Ltd.,  
New York, New York 10017  
and  
Society of the Plastics Industry, Inc.  
New York, New York 10017**

**U.S. DEPARTMENT OF COMMERCE  
Robert A. Mosbacher, Secretary  
NATIONAL INSTITUTE OF STANDARDS  
AND TECHNOLOGY  
John W. Lyons, Director**

**NIST**



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# **REDUCTION OF HYDROGEN CYANIDE CONCENTRATIONS AND ACUTE INHALATION TOXICITY FROM FLEXIBLE POLYURETHANE FOAM COMBUSTION PRODUCTS BY THE ADDITION OF COPPER COMPOUNDS**

## **PART III. THE EFFECT OF COPPER ADDITIVES ON THE FLAMMABILITY CHARACTERISTICS OF FLEXIBLE POLYURETHANE FOAM<sup>1</sup>**

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### **ABSTRACT**

Previous reports from this laboratory have shown that flexible polyurethane foams (FPU) treated with copper dust, cupric oxide, cuprous oxide or copper sulfate produced significantly less hydrogen cyanide (HCN) when thermally decomposed than the identical but untreated control foams. The decreased atmospheric concentrations of HCN resulted in the reduction of the acute inhalation toxicity (as measured by lethality in Fischer 344 rats) produced from exposure to this smoke. This reduction of HCN and toxicity occurred regardless of whether the copper or copper compound was added to the foam during its formulation (prior to the foaming process) or as a post-treatment (after formulation). In all these reported experiments, the foams were thermally decomposed in the NBS Toxicity Test Method apparatus via a two phase procedure previously shown to produce high concentrations of HCN.

This report addresses the issue of whether the addition of a copper compound to a flexible polyurethane foam would affect the flammability characteristics of the foam. The following properties were examined: 1. ignitability in three systems [the NBS Toxicity Test Method, the Cone Calorimeter, and Lateral Ignition and Flame Spread Test (LIFT)], 2. heat release rate under small-scale (Cone Calorimeter) and medium-scale (furniture calorimeter), 3. smoke obscuration (Cone Calorimeter), and 4. rate of flame spread (LIFT). In all cases, no differences in flammability characteristics between the treated and untreated foam were observed.

Key words: acute toxicity; combustion products; copper; flammability; flexible foams; polyurethane foams; hydrogen cyanide; ignitability; inhalation; heat release rate; flame spread rate; smoke opacity.

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<sup>1</sup>This report is a contribution of the National Institute of Standards and Technology and is not subject to copyright.

## 1.0 INTRODUCTION

Eighty percent of fire deaths are from smoke inhalation and not from burns [1]<sup>2</sup>. Carbon monoxide (CO), hydrogen cyanide (HCN), carbon dioxide (CO<sub>2</sub>), and reduced oxygen (O<sub>2</sub>) are the major gases contributing to these smoke inhalation deaths. The most common scenario leading to fire deaths begins with a cigarette which is inadvertently dropped on an upholstered sofa or chair [2]. In this scenario, the furniture, which contains flexible polyurethane foam padding, smolders for a while and then bursts into flames. Previous studies in this laboratory have shown that significantly increased amounts of HCN are generated from flexible polyurethane foam if the material is exposed to a two-phase thermal decomposition which simulates the smoldering and then flaming observed in real upholstered furniture fires [3]. In phase one, the foam is heated at a non-flaming temperature slightly below its autoignition temperature. A char forms. In phase two, the temperature of the charred material is increased and if enough material is present, the flexible polyurethane will undergo a transition to flaming. It is at this smoldering-flaming transition or when the charred material is heated that increased quantities of HCN are generated.

Most analytical chemists are aware that one should not use copper tubing on instruments that are involved in the measurement of HCN because the interaction between the copper and the cyanide interferes with the measurement. Based on this information, we decided to test whether the addition of copper to a flexible polyurethane foam would lower the atmospheric concentration of cyanide normally produced when the foam is thermally decomposed. In our preliminary experiments [4], a copper or cupric oxide (CuO) slurry in water or a copper sulfate (Cu<sub>2</sub>SO<sub>4</sub>) solution was squeezed through a two inch cubic piece of foam. After drying, the foam was thermally decomposed via the two-phase procedure described above. The copper- or copper compound-treated foam produced ninety percent less cyanide than a control foam which was treated only with water. The acute inhalation toxicity as measured by the lethality of rats in the NBS Toxicity Test Method was also reduced.

To determine if the reduction in HCN and acute toxicity would occur if the copper or copper compounds were added during the formulation of the foam rather than as a post-treatment additive, five foams were prepared by a raw materials supplier to the polyurethane industry. Two different concentrations of copper or cuprous oxide (Cu<sub>2</sub>O) were mixed in prior to the foaming process. The foams tested were untreated (control foam) or treated with 0.007% Cu, 0.07% Cu, 0.007% Cu<sub>2</sub>O, or 0.07% Cu<sub>2</sub>O. Small-scale laboratory experiments similar to those conducted with the post-treated foams plus examination of the animals blood cyanide levels following exposures to the combustion products from the various foams were performed [5].

These experiments agreed with earlier work by Jellinek and coworkers who found that the concentrations of HCN generated from the thermal decomposition of a polyurethane at 300 and 400°C decreased when flowed through copper compounds [6,7]. (In our experiments, the temperature of phase 1 was 375°C and that of phase 2 was increased approximately 13°C/min. until 800°C was reached.) The polyurethane films examined by Jellinek were usually 15µm thick (50 mg). In some experiments, the metal powder was mixed with the polymer and, in others, copper metal films of 400 to 1000 Å were deposited on top of the polymer films. In most cases,

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<sup>2</sup> Numbers in brackets refer to references listed at the end of this document.



the percent of copper was 10% or greater. The lowest concentration that they tested was a 2.6% copper film which inhibited the evolution of HCN by 66%. Their experiments indicated that copper acts as an oxidative catalyst which will decompose gaseous HCN into  $N_2$ ,  $CO_2$ ,  $H_2O$  and small amounts of nitrogen oxides. They did not examine the issue of toxicity.

From our first two years of work [4,5,8] sponsored by the International Copper Association, Ltd., we concluded that:

- Hydrogen cyanide concentrations in the thermal decomposition products from a flexible polyurethane foam were significantly reduced when the foam was treated with copper or copper compounds and thermally decomposed via a two-phase system in the NBS Toxicity Test Procedure.
- Significant reduction of HCN concentrations occurred at a concentration of copper (from the addition of cupric oxide) of 0.08% by weight or cuprous oxide of 0.07% by weight.
- Blood cyanide levels in animals exposed to combustion products from the cupric oxide treated foams for 30 minutes were reduced at least half that measured in the animals exposed to the smoke from the same amount of untreated foam.
- The within-exposure deaths as expected from the HCN yields were reduced by the addition of copper and copper compounds.
- Post-exposure deaths appeared to be unaffected by the combustion products from the cupric oxide treated foams. (Note: these delayed post-exposure deaths have not been observed in animals exposed to combustion products from flexible polyurethane foams decomposed in large-scale room fire tests. The cause of these post-exposure deaths is not known.) However, the cuprous oxide treated foams did produce fewer post-exposure deaths than the cupric oxide treated foams or the untreated foams.
- The copper or copper compounds could be added to the foams during or after the foams were formulated and still be effective in reducing the toxicity and HCN yield under the specified test conditions.

In the present study, sponsored by the International Copper Association, Ltd. and The Society of the Plastics Industry, Inc., the following flammability characteristics were examined:

1. Ignitability in three systems [the NBS Toxicity Test Method [9], the Lateral Ignition and Flame Spread Test (LIFT) [10], and the Cone Calorimeter [11]],
2. Heat release rate, heat of combustion and CO/CO<sub>2</sub> ratios under small-scale (Cone Calorimeter) and medium-scale (furniture calorimeter) conditions,
3. Smoke obscuration (Cone Calorimeter), and
4. Rate of flame spread (LIFT).

The objective was to ensure that the addition of cuprous oxide (the copper compound which was the most efficient at reducing the generation of HCN and acute toxicity among the copper compounds tested in our previous studies) did not adversely affect flammability properties of the flexible polyurethane foam. In addition to the flammability tests, small-scale toxicity tests in the NBS Toxicity Test Method were performed to assure that the foams used in this study responded in a similar manner to those tested previously.

## **2.0 MATERIALS AND METHODS**

### **2.1 MATERIALS**

#### **2.1.1 Foams**

Two sets of foams (with and without cuprous oxide) were prepared by BASF Corporation<sup>3</sup> for this series of tests. The cuprous oxide (Cu<sub>2</sub>O: M.W. 143.08) was obtained from American Chemet Corp., Deerfield, IL. These foams were continuously machine mixed on a pilot scale. The size of the laboratory buns were approximately 102 cm (40 inches) wide by 366 cm (12 feet) long and 51 cm (20 inches) high. They were cut to 102 cm (40 inches) long by 102 cm (40 inches) wide by 10 cm (4 inches) high before being shipped to the National Institute of Standards and Technology (NIST). BASF indicated that the cuprous oxide imparted a tan color to the foam and resulted in faster creaming of the reaction. We found that the copper-treated foams

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<sup>3</sup>Certain commercial equipment, instruments, materials or companies are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment or material identified is necessarily the best available for the purpose.

appeared to have a slight pink color. There was no evidence of scorching in these laboratory size buns. The formulations are given in Table 1.

Table 1

Formulation of the Untreated and Cu<sub>2</sub>O-Treated Foams

INGREDIENTS	CONTROL FOAM (No Cu <sub>2</sub> O) (%)	FOAM (Cu <sub>2</sub> O) (%)
Polyoxypropylene polyol (3000 M.W.)	65.6	65.6
Water	2.3	2.3
Silicone surfactant	0.7	0.7
Amine catalyst	0.2	0.2
Tin catalyst	0.3	0.3
TDI (110 index)	30.9	30.9
Cu <sub>2</sub> O	0	0.1

For the various flammability and toxicity tests conducted for this report, the necessary size samples were cut from the larger pieces of foam.

#### 2.1.2 Calibration gases

The calibration gases for the CO, CO<sub>2</sub>, and HCN measurements were commercially supplied in specified concentrations in nitrogen. The concentration of HCN in the commercially supplied cylinders was routinely checked by silver nitrate (AgNO<sub>3</sub>) titration [12], since it is known that the concentration of HCN stored under these conditions will decrease with time.

#### 2.1.3 Animals

Fischer 344 male rats, weighing 200 to 300 grams, were obtained from Taconic Farms (Germantown, NY). Animal care and maintenance were performed in accordance with the procedures outlined in the National Institutes of Health's "Guide for the Care and Use of Laboratory Animals." Each rat was housed individually in suspended stainless steel cages and provided with food (Ralston Purina Rat Chow 5012) and water *ad libitum*. Twelve hours of fluorescent lighting per day were provided using an automatic timer. The rats were allowed to acclimate to laboratory conditions for at least seven days prior to experimentation.



## 2.2 IGNITABILITY

Ignitability of the treated and untreated flexible polyurethane foams was examined in three different systems - the NBS toxicity test method [9], the Cone Calorimeter [11] and the LIFT apparatus [10]. In the NBS toxicity test method, the materials' autoignition temperatures may be used to compare the propensity of the materials to ignite. In this case, the autoignition temperature is defined as the lowest furnace temperature at which a material sample introduced into the test furnace will spontaneously ignite within 30 minutes without the help of any igniter or sparker. To determine the autoignition temperature, the temperature of the furnace is set at 500°C and when this temperature is attained, approximately one gram samples are introduced into the furnace. If autoignition does not occur, the process is repeated at increasing 50°C intervals until the material does flame. The autoignition temperature should be finally determined within 25°C. If autoignition does occur at 500°C, the furnace temperature is decreased in increments until the autoignition temperature is bracketed within 25°C. The sparker which is used in flaming experiments to assure immediate flaming is not used for the determination of the autoignition temperature. Since the autoignition temperature is dependent on sample size, the sample size should be increased to the maximum that one anticipates using for the toxicity tests and checked to assure the larger size will not ignite at a lower temperature. When the autoignition temperature of the material is established, the furnace temperature is decreased by 25°C for testing the non-flaming condition (in these experiments, the temperature used in phase 1 was approximately 375°C). As the determination of the autoignition temperature depends upon the apparatus and procedure, the autoignition temperatures determined by this procedure may differ from those measured according to ASTM D 1929.

In the Cone Calorimeter, ignitability was determined with a sample size of 10 cm long x 10 cm wide x 5 cm thick. All sides except the top were covered with aluminum foil. The sample was placed in the horizontal orientation and the igniter was placed 20 mm above the surface of the material. The ignitability of the sample was then examined at different fluxes and the time of ignition and the flux at which no ignition occurred was noted. At low fluxes, the continuous use of the igniter contributed to the radiation that the material received; therefore, in those cases, the igniter was used in an intermittent mode in which the sparker would be on for approximately 0.7 seconds and then off for the same amount of time. The material was exposed to the intermittent spark igniter for 10 minutes or until ignition. The lowest flux that produced ignition within the 10 minutes was considered the critical ignition for flux of the material.

The LIFT Apparatus [10] can be used to determine the minimum surface flux and the effective temperature necessary for ignition. The main components of this apparatus are a pre-mixed natural gas-air radiant heat source (which is set at a 15 degree incline to the sample), a sample holder, and an applied pilot flame to promote ignition. The sample size used to determine ignitability was 155 mm long x 155 mm wide by 30 mm thick. The sides and back of each sample as well as approximately 6 mm of the surface edges were covered with aluminum foil. Due to the thickness limitation of the specimen holder, a 12 mm Marinite non-combustible board with a density of  $750 \pm 100 \text{ Kg/m}^3$  was used as the specimen backing. The specimen holder shown in Figure 1 has a contiguous wall flange that is positioned above the sample to facilitate the development of a boundary layer containing pyrolyzed gases and entrained room air. An acetylene-air pilot flame is positioned 1 cm above the sample surface and parallel to the wall flange such that it interrupts the gaseous mixture containing the pyrolyzed gases generated at the sample surface. After the air-gas fueled radiant panel was set to the desired flux level (this



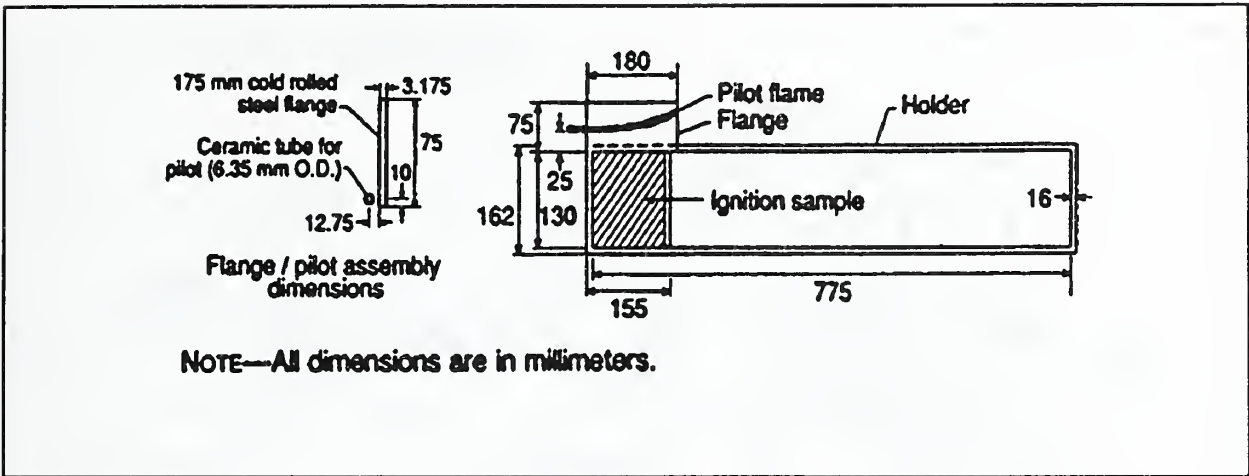


Figure 1. Specimen holder and pilot configuration for ignition test

flux includes the contribution of the continuous pilot), the sample was vertically mounted and exposed to the radiant heat (Fig. 2). The time to ignition (using piloted ignition) was determined at various fluxes (13 to 26 kW/m<sup>2</sup>) and the critical flux for ignition was extrapolated from the curve of the data. The data from the critical radiative heat flux is then used to infer the surface ignition temperature from the following equation:

$$\dot{q}_{o,ig}'' = h_c(T - T_\infty) + \sigma(T^4 - T_\infty^4) - h(T_{ig} - T_\infty)$$

where a black surface is assumed and  $\dot{q}_{o,ig}''$  is the critical flux for ignition (kW/m<sup>2</sup>),  $h_c$  is the convective heat loss coefficient,  $h$  is the overall heat loss coefficient (kW/m<sup>2</sup>K),  $T$  is the surface temperature (K),  $T_\infty$  is the ambient and initial temperature (K),  $\sigma$  is the Stefan-Boltzman constant (kW/m<sup>2</sup>K<sup>4</sup>), and  $T_{ig}$  is the ignition temperature (K).

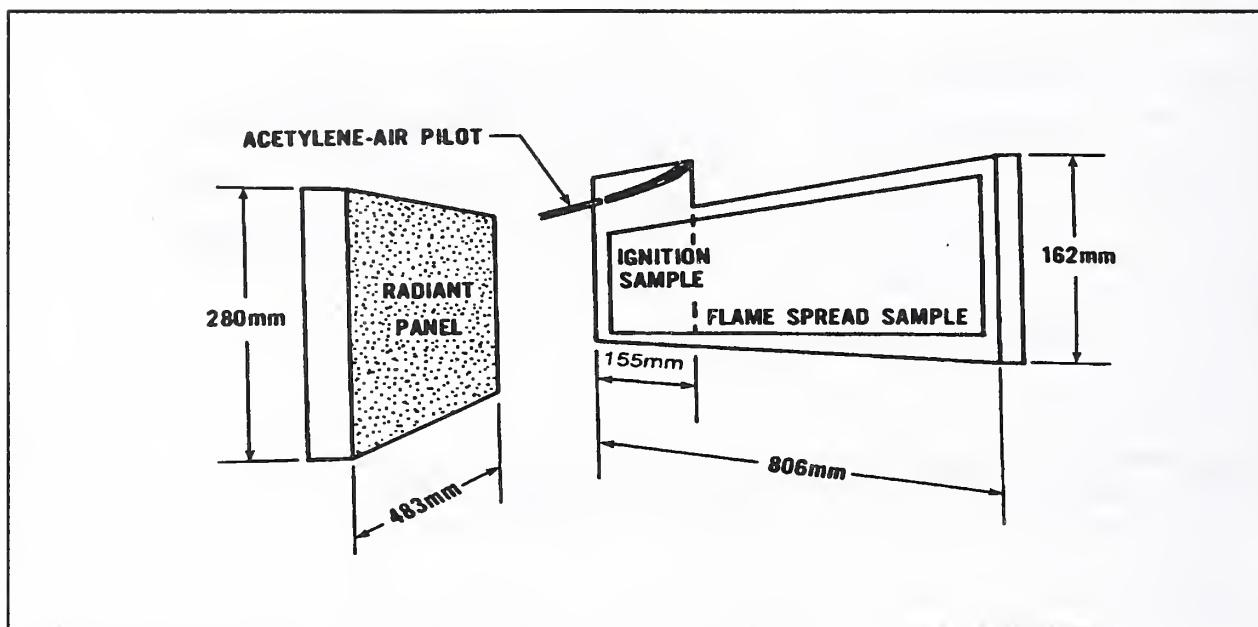


Figure 2. Schematic of LIFT apparatus with ignition sample

## 2.3 HEAT RELEASE RATE

### 2.3.1 Cone Calorimeter

The rate of heat release was measured in the NBS Cone Calorimeter (Figure 3) [11]; this procedure has recently been published as an ASTM standard test method. Briefly, this bench-scale instrument determines the heat release rate by measuring the oxygen depletion during the flaming combustion of a material. An external radiant flux as high as  $100 \text{ kW/m}^2$  may be imposed on the specimen by a temperature-controlled electric heater shaped like a cone. Since the heater operates at a moderate temperature (up to  $1000^\circ\text{C}$ ) and behaves very nearly as a black body, the effective spectral distribution is likely to be very close to that expected from compartment fires [13].

In these experiments, specimens were tested only in the horizontal position to avoid melting and dripping problems. The sample size was 10 cm long x 10 cm wide x 5 cm thick and all sides except the top were covered with aluminum foil. The samples were exposed to a preset external irradiance with the spark igniter mounted 13 mm above the center of the sample to ignite the pyrolysis products. Sparking was initiated at the beginning of the exposure and applied in the continuous mode until sustained burning developed across the sample surface. Changes in sample mass are measured continuously by a load cell. Oxygen concentrations are measured by a paramagnetic analyzer with a range from 0 to 25%. Carbon monoxide (CO) and carbon dioxide ( $\text{CO}_2$ ) are measured by non-dispersive infrared analyzers. Calculations to determine rate of heat release and heat of combustion are provided in reference 11. Tests were terminated when flaming on the sample extinguished. Three replicate experiments with each foam were conducted at each of three external irradiances: 10, 15, and  $35 \text{ kW/m}^2$ . At  $35 \text{ kW/m}^2$ , ignition

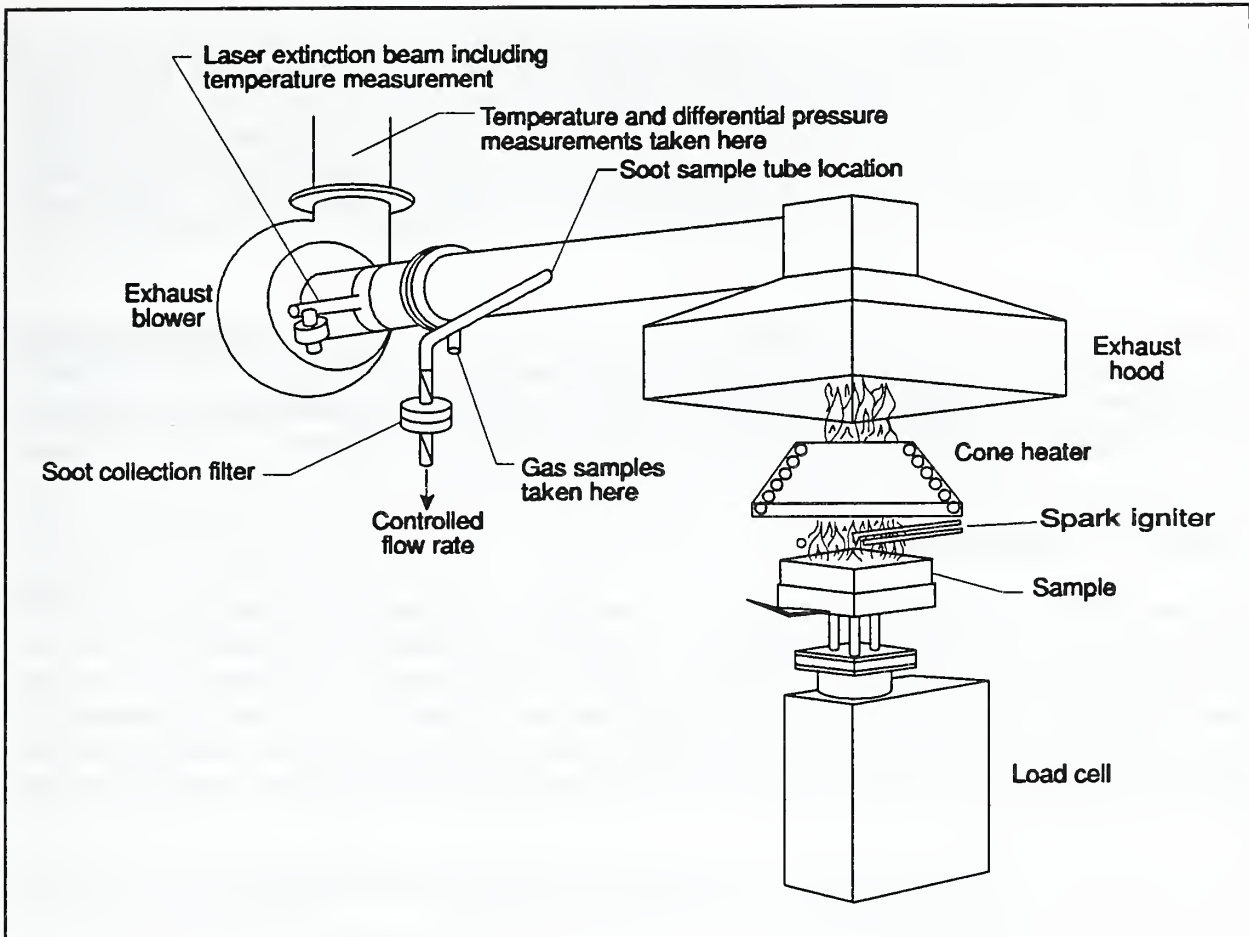


Figure 3. Schematic of the Cone Calorimeter

was virtually instantaneous for both foams. Therefore, tests were conducted at lower external irradiances in order to examine the two foams under conditions which were expected to show differences in the various flammability factors under study.

### 2.3.2 Furniture calorimeter

The furniture calorimeter [14] was designed to measure the heat release and mass loss rates of furniture items burning in the open air. The basic principle of the apparatus, oxygen consumption calorimetry, is the same as that of the Cone Calorimeter previously described. The heat release rate is computed from measurements of mass flow and oxygen concentration through the exhaust stack. The effective heat of combustion can readily be determined from the heat release rate and the corresponding measured mass loss rate of the test sample. Carbon monoxide and  $\text{CO}_2$  concentrations also were monitored during each test and used to correct the heat release rate calculations; detailed calculations for making these corrections are described by Parker [15].

Test samples were composed of two blocks of foam, each measuring 1.0 m by 0.5 m by 0.1 m thick. The foam blocks were stacked to form a 0.2 m thick polyurethane foam structure. A specially designed electric heater was placed between the two foam blocks. This heater system was used to initiate nonflaming combustion. Approximately one hour after initiation of nonflaming combustion, a small propane flame was applied to the charred material to initiate flaming combustion. Two replicates of each foam (treated and untreated) were tested. During two of the four tests, one with a copper treated foam and one with a nontreated foam, the samples self-ignited.

The electric heater assembly was made by evenly winding 2.33 m of # 28 nichrome wire around two 3 mm diameter ceramic rods for a total electrical resistance of 34.5 ohms. By trial and error, it was found that an applied voltage of 65-70 VAC would provide sufficient thermal energy to cause the foams to smolder without forcing them into flaming combustion.

## 2.4 RATE OF FLAME SPREAD

The rate of flame spread of the treated and untreated foams was measured using the LIFT apparatus [16,17]. This test determines the velocity of the lateral flame spread on a vertical surface under a specified graduated radiant heat flux. This laboratory-scale device has recently been published as an ASTM standard [10]. The main components of this apparatus are described in section 2.2. A schematic of the apparatus is shown in Figure 2 and the external irradiance distribution at the sample surface, normalized to its value at 50 mm, is shown in Figure 4.

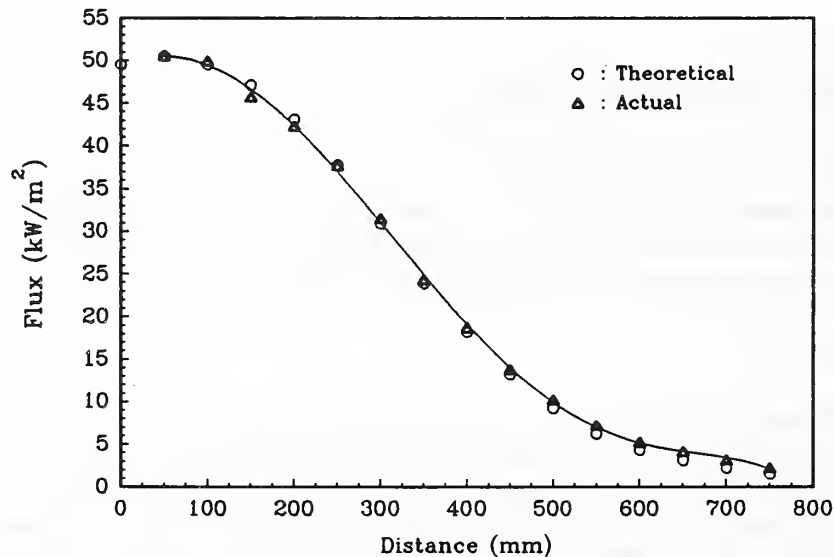


Figure 4. Flux distribution along specimen surface.

Flame spread tests were conducted as described by Quintiere and Harkleroad [16] with sample sizes of 155 X 800 mm. The samples were covered with aluminum foil on all sides except the



surface. The specimens were exposed to an external irradiance that was approximately 5 kW/m<sup>2</sup> higher at the hot end than the minimum external irradiance required for ignition. The samples were preheated at this radiant flux until a state of thermal equilibrium was achieved; based on an analysis of the time-to-ignition data, the preheat time was 38 seconds. After the ignition of the sample by the external pilot, the movement of the flame front across the horizontal surface is followed as a function of time.

## 2.5 SMOKE OBSCURATION

The amount of smoke produced in a fire can interfere with a person's ability to see and, therefore, to escape. Thus, it is important to determine if smoke formation changed in the presence or absence of Cu<sub>2</sub>O. Smoke obscuration was determined with the Cone Calorimeter, which is equipped with a helium-neon laser located in the exhaust duct downstream of the burning sample and which measures the extinction of light from the smoke [18]. The specific extinction area (m<sup>2</sup>/kg) is given by:

$$\sigma_f = k \frac{\dot{V}}{\dot{m}}$$

where k is the light extinction coefficient (m<sup>-1</sup>) and is defined by:

$$k = \frac{1}{L} \ln \left( \frac{I_o}{I} \right)$$

where L is the beam path length through the measurement space (m); I<sub>o</sub> is the initial light intensity; I is the final light intensity; V is the flowrate in the exhaust duct (m<sup>3</sup>/s); and  $\dot{m}$  is the mass loss rate of the sample (kg/s). The values given are the average of each test.

These measurements were taken simultaneously with the heat release rate measurements. Therefore, the samples were treated as described previously in section 2.3.1. Each foam was tested three times at each flux - 10, 15, and 35 kW/m<sup>2</sup>.

## 2.6 SMALL-SCALE TOXICITY TESTS (ANIMALS)

To assure that these newly prepared  $\text{Cu}_2\text{O}$ -treated and untreated foams reacted in a similar fashion to those tested previously in our laboratory, the HCN generation and the acute inhalation toxicity of the combustion products of each of the two sets of foams was assessed by the NBS Toxicity Test Method [9]. This method was modified to provide a two phase heating regime to simulate the common fire scenario in which a chair smolders for some time before flaming [3]. In this procedure, the material was first heated in a non-flaming mode for 30 minutes at approximately  $25^\circ\text{C}$  below the predetermined autoignition temperature ( $400^\circ\text{C}$ ) of the control foam. If flaming occurred, the temperature of the non-flaming phase was reduced by approximately  $10^\circ\text{C}$  in subsequent experiments. A black char was formed during this first phase. In phase two, the temperature of the furnace was raised approximately  $13^\circ\text{C}/\text{minute}$  up to  $800^\circ\text{C}$ . In all cases in this series of experiments, there was not enough fuel left after phase one to allow flaming to occur during phase two. The details of the animal exposures can be found in references 4 and 5.

## 3.0 RESULTS AND DISCUSSION

### 3.1 IGNITABILITY

#### 3.1.1 Autoignition Temperatures Determined by the NBS Toxicity Test Method

The autoignition temperature of both the  $\text{Cu}_2\text{O}$ -treated and untreated flexible polyurethane foam used in this series of experiments was found to be  $400^\circ\text{C}$ . The temperature of phase one is  $25^\circ\text{C}$  below this temperature; at this lower temperature, no flaming is expected. Sometimes, however, if the fuel load is high, the material will flame at the lower temperature. In this series of experiments, 12 tests were run at approximately  $375^\circ\text{C}$  with the  $\text{Cu}_2\text{O}$ -treated foam at fuel loads ranging from 18 to  $40\text{ mg}/\ell$ . In only one case (at  $40\text{ mg}/\ell$ ) was flaming observed. In a duplicate experiment at  $40\text{ mg}/\ell$ , no flaming occurred. In the non-treated foam experiments, 16 tests were conducted with fuel loads ranging from 12.5 to  $28\text{ mg}/\ell$  and no flaming was observed. These results indicate that the autoignition temperature of  $400^\circ\text{C}$  is appropriate for both the treated and untreated foams and there is no difference in thermal ignitability based on this test procedure.

#### 3.1.2 Critical Flux and Surface Ignition Temperatures as Determined with the LIFT Apparatus

In the LIFT tests, four experiments were conducted for each of the two foams ( $\text{Cu}_2\text{O}$ -treated and untreated) to determine the extrapolated critical flux for ignition (Table 2). This data is plotted in Figures 5 and 6. Based on this data, the extrapolated critical flux for ignition was  $11\text{ kW}/\text{m}^2$  for both the treated and untreated foams. This value had to be extrapolated since at the lowest possible flux in the LIFT, the materials would still ignite.

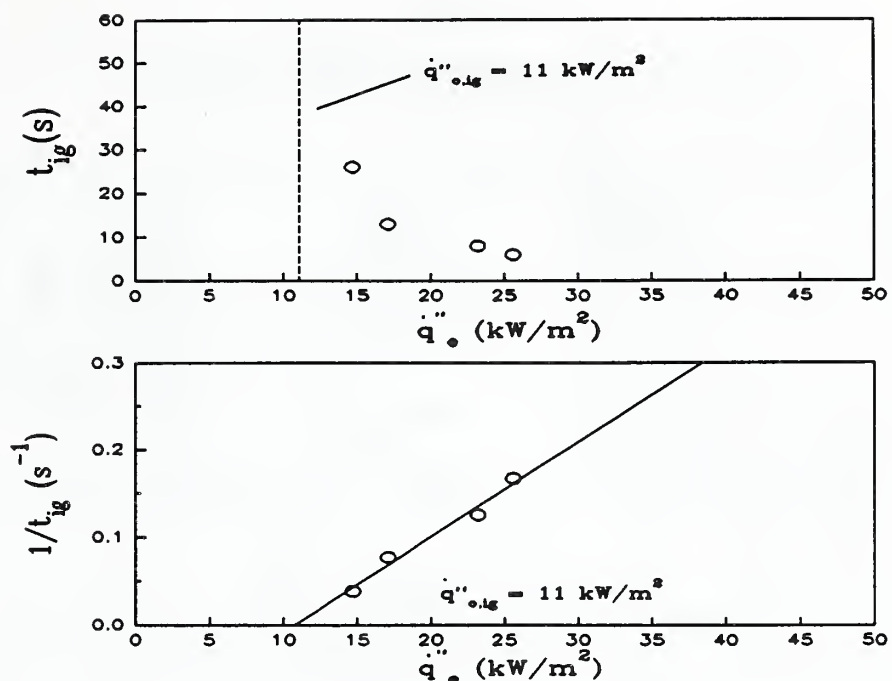


Figure 5. Critical Flux for ignition determined with the LIFT apparatus for the untreated polyurethane foam.

Table 2

Critical Flux for Ignition Determined by the LIFT Apparatus

Polyurethane Foam	Flux ( $\text{kW/m}^2$ )	$1/\text{Time}_{ig}$ (1/s)
Untreated	14.7	0.038
	17.1	0.076
	23.2	0.128
	25.6	0.172
$\text{Cu}_2\text{O}$ -treated	13.3	0.037
	17.3	0.077
	23.8	0.159
	26.1	0.217

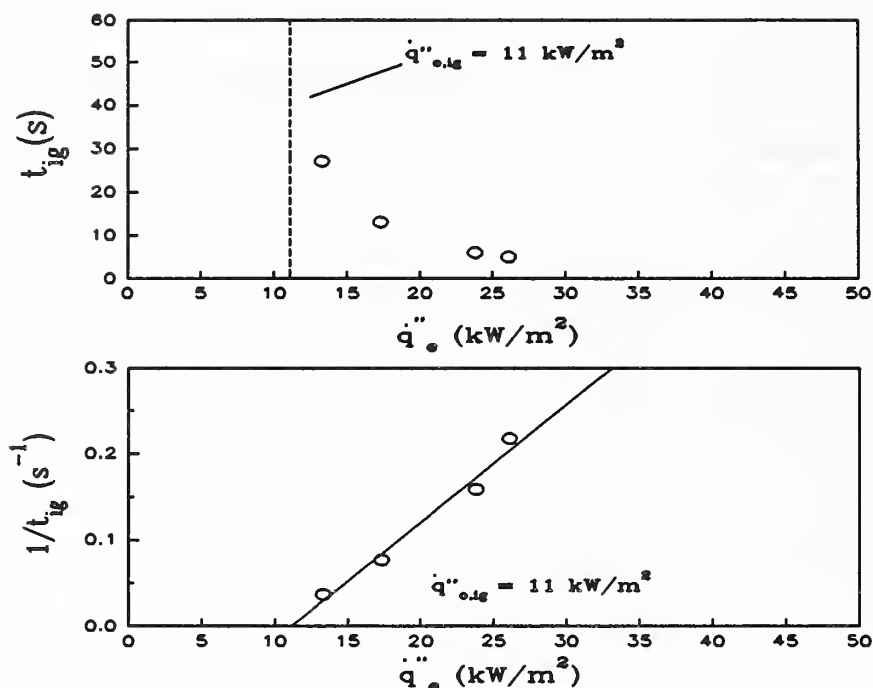


Figure 6. Critical flux for ignition determined by the LIFT apparatus for the  $\text{Cu}_2\text{O}$ -treated foam.

Based on the equation in section 2.2, the surface temperature at ignition of both the untreated foam and the  $\text{Cu}_2\text{O}$ -treated foam was  $320^\circ\text{C}$ .

### 3.1.3 Lowest Flux for Ignition as Determined by the Cone Calorimeter

The ignitability of the two foams as determined by the Cone Calorimeter was examined at 5 different heat fluxes - 35, 15, 10, 9, and  $8 \text{ kW/m}^2$ . As the flux increased, the time to ignition decreased (Table 3). At the two lowest fluxes, an intermittent igniter as described in section 2.2 was used to ensure that the radiation on the sample surface was not influenced by the igniter itself. For both foams, the lowest flux which still caused ignition of the samples was  $9 \text{ kW/m}^2$ .



Table 3

Determination of the Lowest Flux Producing Ignition of  
Treated and Untreated Foams in the Cone Calorimeter

Flux (kW/m <sup>2</sup> )	Untreated Foam Ignition Time (s)	Mean Ignition Time $\pm$ S.D. (s)	Cu <sub>2</sub> O-Foam Ignition Time (s)	Mean Ignition Time $\pm$ S.D. (s)
35 <sup>a</sup>	Immediate <sup>b</sup>		Immediate	
	Immediate	Immediate	Immediate	Immediate
	Immediate		Immediate	
15 <sup>a</sup>	12.1		10.1	
	12.1	12.2 $\pm$ 0.1	14.8	12.0 $\pm$ 2.5
	12.3		11.2	
10 <sup>a</sup>	52.0		45.6	
	43.7	45.4 $\pm$ 5.9	49.3	46.5 $\pm$ 2.5
	40.5		44.6	
9 <sup>c</sup>	85.0		79.0	
	114.0	99.5	79.0	75.6 $\pm$ 5.8
	no ignition		69.0	
8 <sup>c</sup>	no ignition		no ignition	

a. Igniter applied in a continuous mode

b. Sample flamed at time igniter started (less than 3 seconds)

c. Igniter applied in an intermittent mode.

### 3.2 HEAT RELEASE RATE

#### 3.2.1 Cone Calorimeter

Triplicate tests were performed for each foam at each of three heat fluxes - 10, 15, and 35 kW/m<sup>2</sup>. Tables 4 to 6 provide the results on peak rate of heat release, heat of combustion and average CO/CO<sub>2</sub> ratios for each test.

Table 4

Comparison of Peak Heat Release Rates of Untreated and  $\text{Cu}_2\text{O}$ -Treated Foams

Flux (kW/m <sup>2</sup> )	Untreated Peak RHR <sup>a</sup> (kW/m <sup>2</sup> )	Mean $\pm$ S.D. Untreated Peak RHR (kW/m <sup>2</sup> )	$\text{Cu}_2\text{O}$ -foam Peak RHR (kW/m <sup>2</sup> )	Mean $\pm$ S.D. $\text{Cu}_2\text{O}$ -foam Peak RHR (kW/m <sup>2</sup> )
10	305	295 $\pm$ 26	289	288 $\pm$ 13
	265		301	
	315		275	
15	299	323 $\pm$ 32	392	354 $\pm$ 40
	312		312	
	359		357	
35	601	534 $\pm$ 59	608	557 $\pm$ 46
	488 <sup>b</sup>		518 <sup>c</sup>	
	513		544	

a. Rate of heat release

b. Ignited late - 8 seconds; others at 35 kW/m<sup>2</sup> ignited in approximately 2 to 3 seconds.c. Ignited late - 4 seconds; others at 35 kW/m<sup>2</sup> ignited in approximately 2 to 3 seconds.

Table 5

Comparison of Heat of Combustion Determined in the Cone Calorimeter  
for Cu<sub>2</sub>O-Treated and Untreated Foams

Flux (kW/m <sup>2</sup> )	Untreated Avg. Heat of Combustion (MJ/kg)	Mean $\pm$ S.D. Untreated Avg. Heat of Combustion (MJ/kg)	Cu <sub>2</sub> O-Treated Avg. Heat of Combustion (MJ/kg)	Mean $\pm$ S.D. Cu <sub>2</sub> O-Treated Avg. Heat of Combustion (MJ/kg)
10	26.4	26.5 $\pm$ 0.1	27.1	26.6 $\pm$ 0.5
	26.5		26.7	
	26.6		26.1	
15	26.9	26.5 $\pm$ 0.4	26.5	26.8 $\pm$ 0.4
	26.5		27.3	
	26.2		26.6	
35	27.0	26.5 $\pm$ 1.1	26.4	26.4 $\pm$ 0.2
	27.3		26.2	
	25.3		26.5	

Table 6

Average CO/CO<sub>2</sub> Ratios Determined in the Cone Calorimeter  
for the Untreated and Cu<sub>2</sub>O-Treated Foams

Flux (kW/m <sup>2</sup> )	Untreated CO/CO <sub>2</sub> (kg/kg)	Mean $\pm$ S.D. Untreated CO/CO <sub>2</sub> (kg/kg)	Cu <sub>2</sub> O-Treated CO/CO <sub>2</sub> (kg/kg)	Mean $\pm$ S.D. Cu <sub>2</sub> O-Treated CO/CO <sub>2</sub> (kg/kg)
10	0.0067	0.0067 $\pm$ 0.0009	0.0064	0.0071 $\pm$ 0.0012
	0.0058		0.0085	
	0.0075		0.0063	
15	0.0067	0.0070 $\pm$ 0.0004	0.0061	0.0066 $\pm$ 0.0008
	0.0074		0.0062	
	0.0070		0.0075	
35	0.0075	0.0078 $\pm$ 0.0005	0.0084	0.0087 $\pm$ 0.0003
	0.0083		0.0090	
	0.0075		0.0086	

According to Tables 4 and 5, the peak rates of heat release and the average heats of combustion for both foams at the three tested fluxes are within one standard deviation of the mean. The mean CO/CO<sub>2</sub> ratios are within one standard deviation of each other for the flux levels 10 and 15 kW/m<sup>2</sup>, and within two standard deviations for flux level 35 kW/m<sup>2</sup> (Table 6). The mean and standard deviation of the three sets of heat release data at each tested flux were calculated for each 5 second interval and plotted to illustrate the similarity of heat release response for the two foams (Figs. 7 - 9).

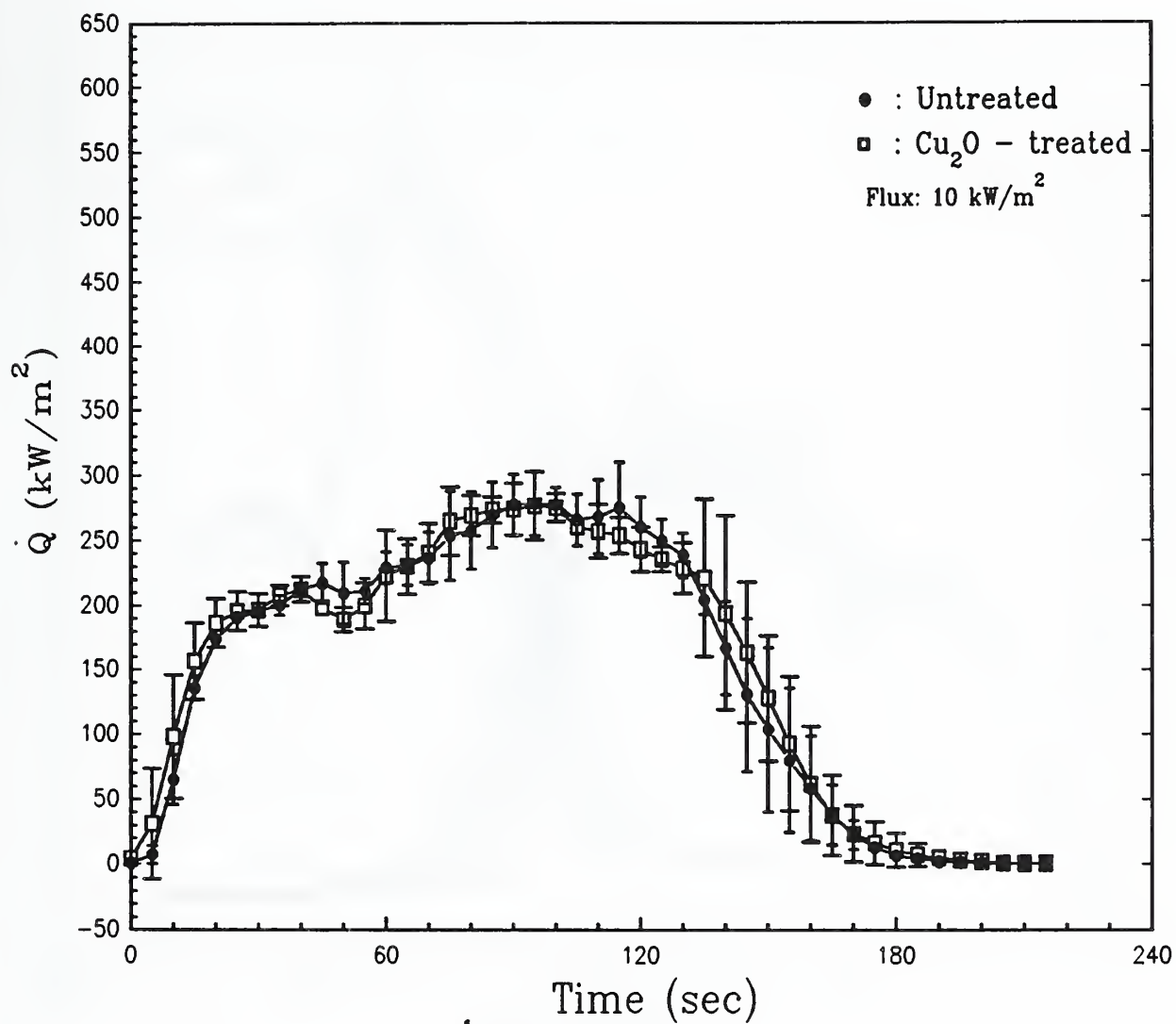


Figure 7. Average heat release rate  $\pm$  standard deviation for three replicate tests of the  $\text{Cu}_2\text{O}$ -treated and untreated flexible polyurethane foam.

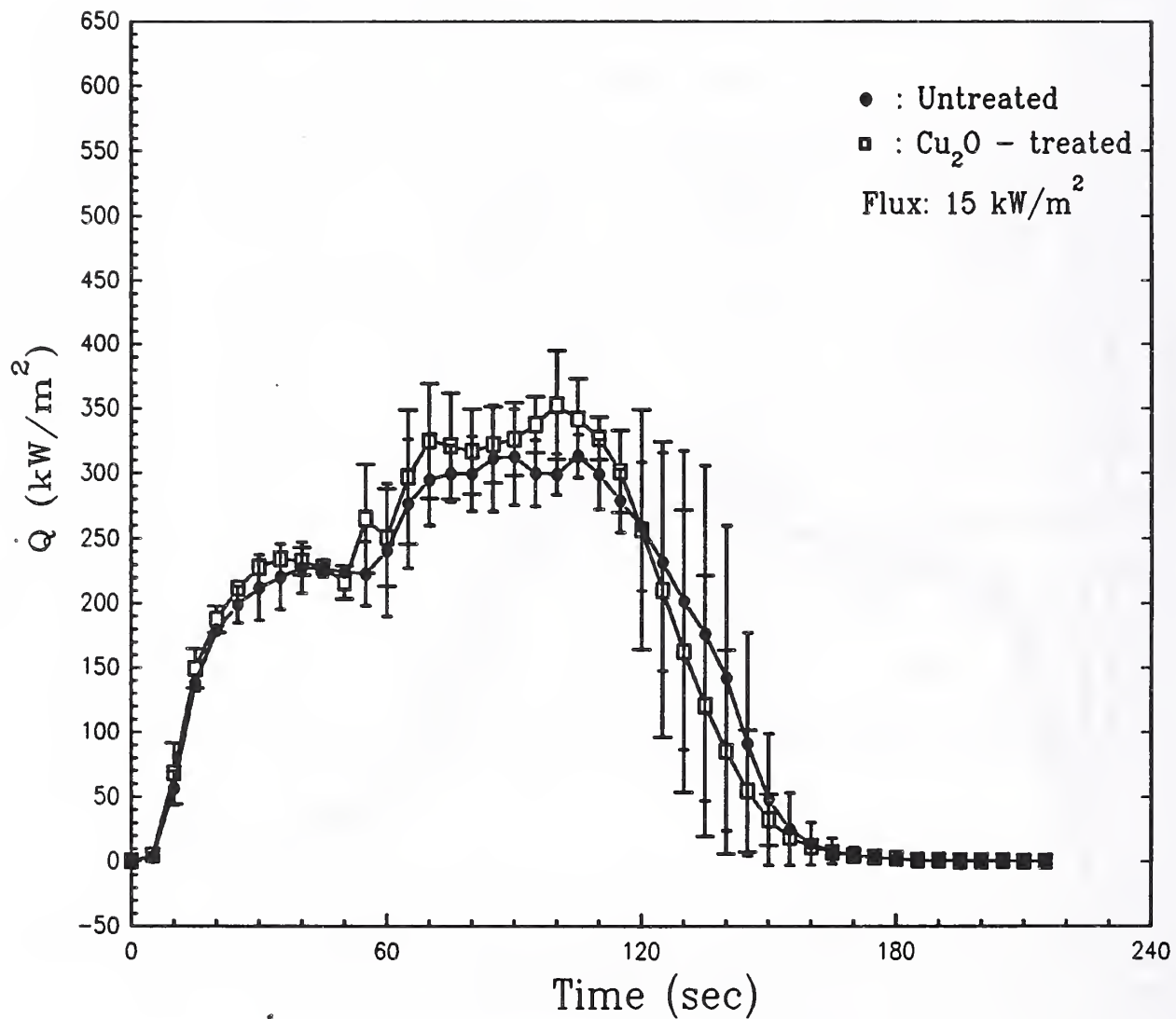


Figure 8. Average heat release rate  $\pm$  standard deviation for three replicate tests of the Cu<sub>2</sub>O-treated and untreated flexible polyurethane foam.

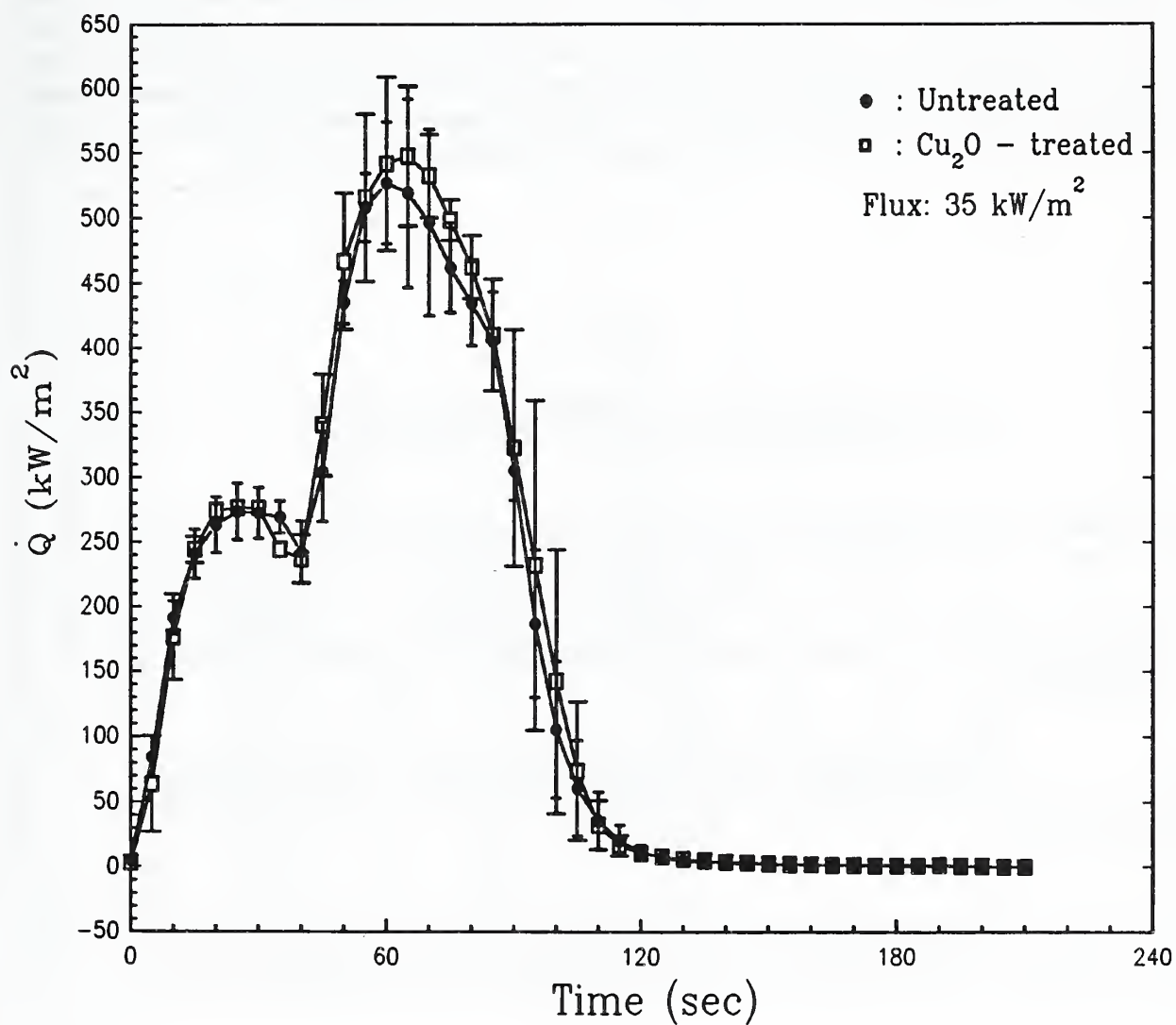


Figure 9. Average heat release rate  $\pm$  standard deviation for three replicate tests of the  $\text{Cu}_2\text{O}$ -treated and untreated flexible polyurethane foam.



### 3.2.2 Furniture calorimeter

Figure 10 shows the rate of heat release for each test and Figure 11 shows the average heat release rate for the replicate tests. Table 7 summarizes the peak heat release rate and the average of two replicates along with the effective heat of combustion and the overall CO/CO<sub>2</sub> ratio after the initiation of flaming combustion. The average peak heat release rate for the copper treated foam was 380 kW and the average peak heat release rate for the untreated foam was 350 kW. The mean effective heats of combustion were 28 MJ/kg and 29 MJ/kg and the mean CO/CO<sub>2</sub> ratios were 0.04 kg/kg and 0.06 kg/kg for the untreated and copper treated foams, respectively. These values and the range of variation between the tests indicate that there was no substantial difference between the burning characteristics of the two foams.

Table 7

Summary of the Results of Furniture Calorimeter Tests on  
Cu<sub>2</sub>O-treated and Untreated Polyurethane Foam

Foam	Test No.	Peak Rate of Heat Release (kW)	Heat of Combustion (MJ/kg)	CO/CO <sub>2</sub> Ratio (kg/kg)
Untreated	1	355	24	0.02
	2	334	31	0.06
	Mean (range)	350 (334-355)	28 (24-31)	0.04 (0.02-0.06)
Cu <sub>2</sub> O-Treated	1	403	28	0.08
	2	355	29	0.04
	Mean (range)	380 (355-403)	29 (28-29)	0.06 (0.04-0.08)



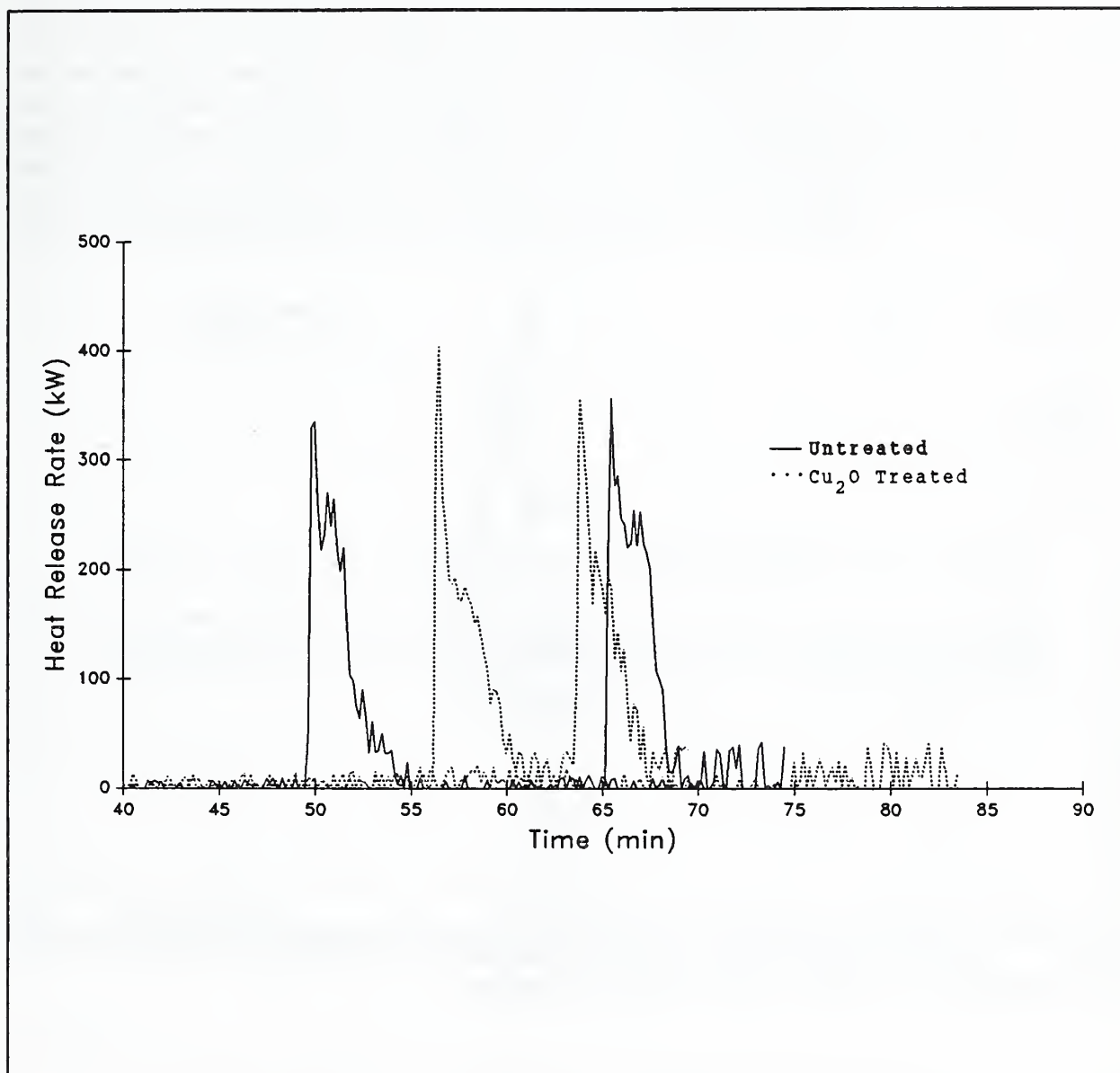


Figure 10. Rate of heat release of individual tests with the furniture calorimeter.

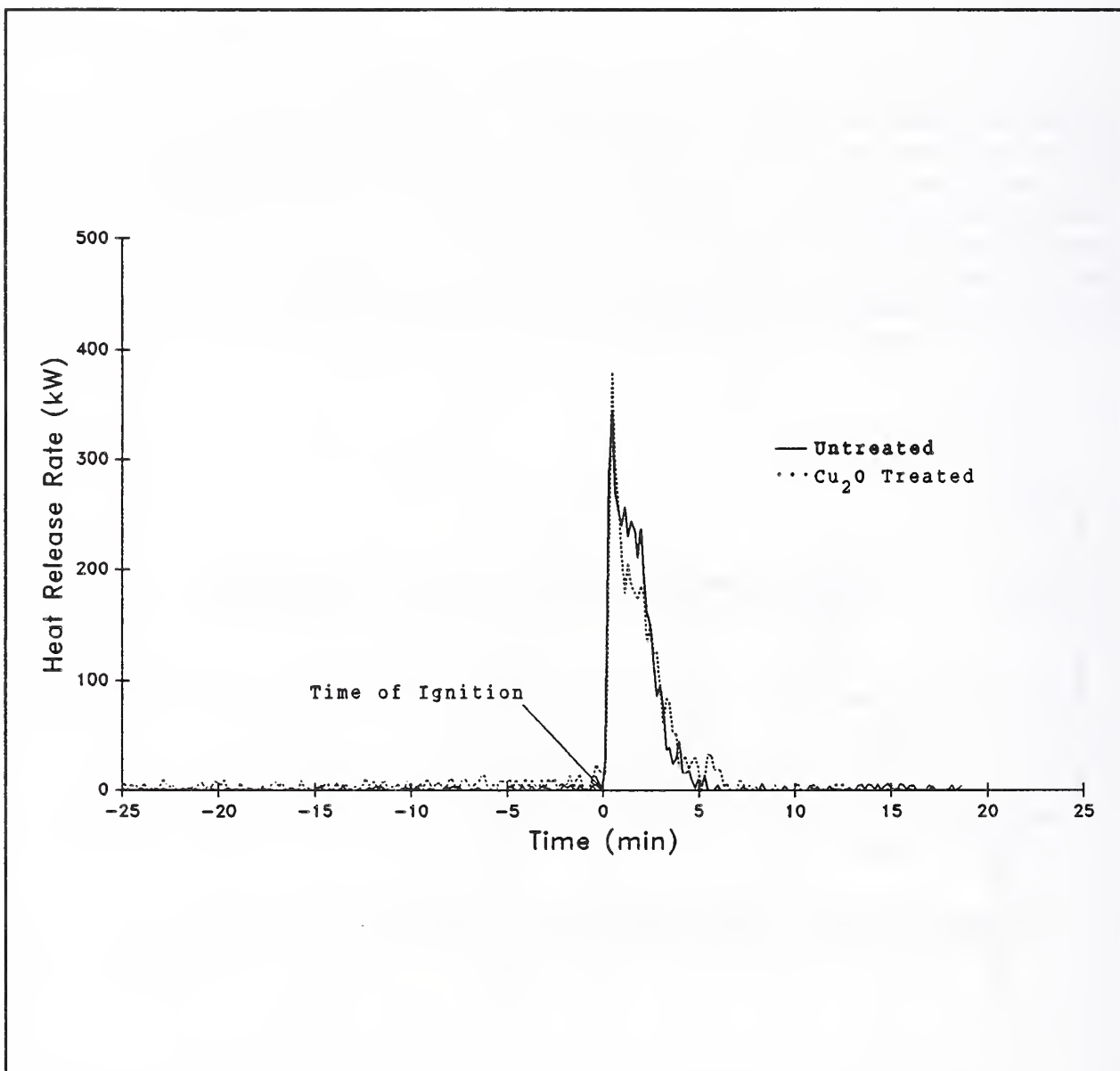


Figure 11. Comparison of the average heat release rate for the two replicate tests of Cu<sub>2</sub>O-treated and untreated polyurethane foam samples.

### 3.3 RATE OF FLAME SPREAD

The rate of flame spread for each foam was determined in three separate experiments. The results of each of these experiments are plotted in Figs. 12 (untreated foam) and 13 ( $\text{Cu}_2\text{O}$ -treated foam). The mean and standard deviation of the regression slopes for the three untreated foam experiments was  $0.057 \pm 0.013$ ; whereas that for the  $\text{Cu}_2\text{O}$ -treated foams was  $0.043 \pm 0.001$ . Therefore, there was no significant difference in the rate of flame spread due to the addition of copper to the foams. Another parameter which is determined along with the rate of flame spread is the minimum flux for spread. Table 8 shows the values obtained for the two foams for the three replicate tests and the mean and standard deviation of the results. These values also indicate that there was no difference between the two foams.

Table 8

Minimum Flux for Flame Spread for the Untreated and  $\text{Cu}_2\text{O}$ -Treated Foams

Test number	Untreated Foam Minimum Flux for Spread (kW/m <sup>2</sup> )	$\text{Cu}_2\text{O}$ -Treated Foam Minimum Flux for Spread (kW/m <sup>2</sup> )
1	0.65	0.64
2	0.64	0.63
3	0.62	0.63
Mean $\pm$ S.D.	$0.64 \pm 0.02$	$0.63 \pm 0.01$

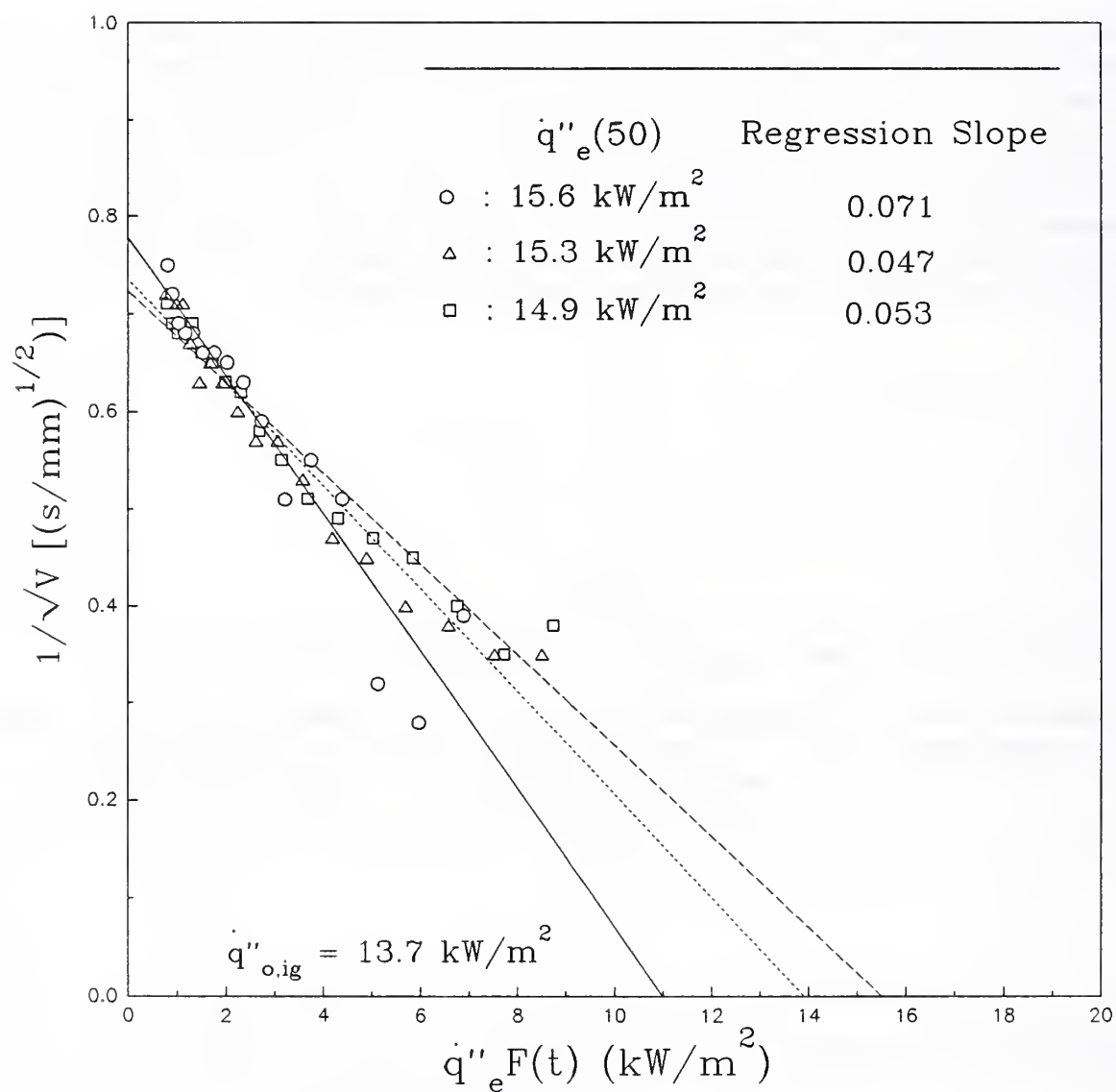


Figure 12. Slopes of regressions for untreated polyurethane foam.

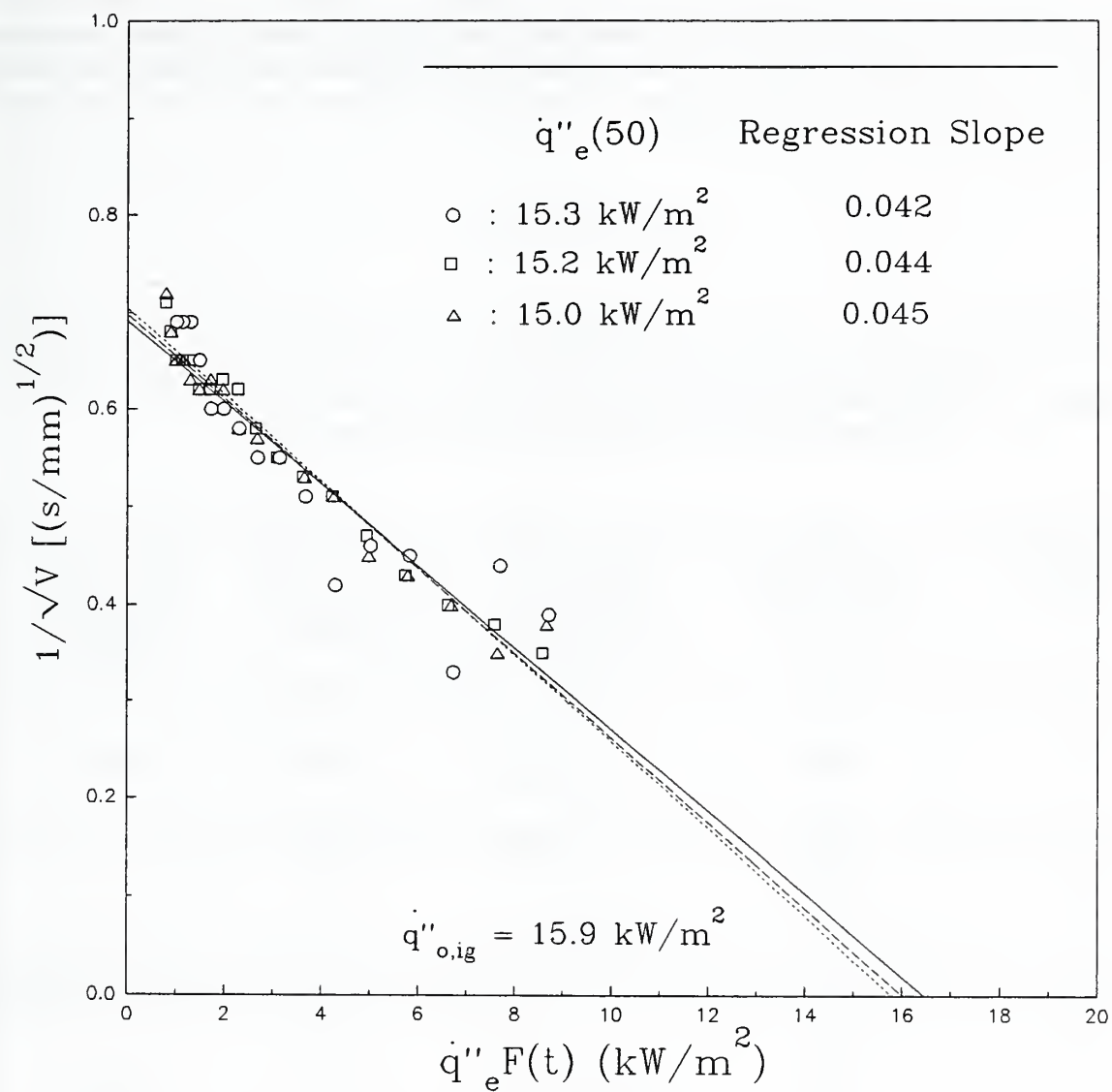


Figure 13. Slopes of regressions for Cu<sub>2</sub>O-treated foam.

### 3.4 SMOKE OBSCURATION

Smoke obscuration was determined with the Cone Calorimeter. Each foam was tested three times at each flux - 10, 15, and 35 kW/m<sup>2</sup> - and the mass weighted average specific extinction area (which is a measure of the smoke obscuration) results are presented in Table 9 along with their means and standard deviations. These results indicate no difference in smoke obscuration between the treated and untreated foams.

Table 9

Comparison of Mass Weighted Average Specific Extinction Areas Generated by the Cone Calorimeter for the Untreated and Cu<sub>2</sub>O-Treated Foams

Flux (kW/m <sup>2</sup> )	Untreated Sp. Ext. Area <sup>a</sup> (m <sup>2</sup> /kg)	Mean $\pm$ S.D. Untreated Sp. Ext. Area (m <sup>2</sup> /kg)	Cu <sub>2</sub> O-Treated Sp. Ext. Area (m <sup>2</sup> /kg)	Mean $\pm$ S.D. Cu <sub>2</sub> O-Treated Sp. Ext. Area (m <sup>2</sup> /kg)
10	189	187 $\pm$ 4	194	189 $\pm$ 5
	182		185	
	190		188	
15	218	218 $\pm$ 3	206	214 $\pm$ 7
	215		219	
	220		218	
35	239	246 $\pm$ 20	245	242 $\pm$ 9
	268		250	
	230		232	

a. Mass Weighted Average Specific Extinction Area taken at 180 seconds

### 3.5 SMALL-SCALE TOXICITY TESTS

The results of the small-scale toxicity tests were similar to those found in tests conducted in this laboratory previously. These similar results indicate that the foams used for this series of tests were not significantly different from those foams used in prior testing.

### 4.0 CONCLUSIONS

Untreated and  $\text{Cu}_2\text{O}$ -treated flexible polyurethane foams were studied to determine if the presence of copper had any effect on the following flammability characteristics:

- Ignitability
- Rate of heat release
- Rate of flame spread
- Smoke obscuration
- Heat of combustion and
- Average  $\text{CO}/\text{CO}_2$  ratios.

In all cases, no significant differences were observed between the foams with and without 0.1% (by weight)  $\text{Cu}_2\text{O}$ .

## 5.0 ACKNOWLEDGEMENTS

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10. SUPPLEMENTARY NOTES

☐ DOCUMENT DESCRIBES A COMPUTER PROGRAM; SF-185, FIPS SOFTWARE SUMMARY, IS ATTACHED.

11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

Previous reports from this laboratory have shown that flexible polyurethane foams (FPU) treated with copper dust, cupric oxide, cuprous oxide or copper sulfate produced significantly less hydrogen cyanide (HCN) when thermally decomposed than the identical but untreated control foams. The decreased atmospheric concentrations of HCN resulted in the reduction of the acute inhalation toxicity (as measured by lethality in Fischer 344 rats) produced from exposure to this smoke. This reduction of HCN and toxicity occurred regardless of whether the copper or copper compound was added to the foam during its formulation (prior to the foaming process) or as a post-treatment (after formulation). In all these reported experiments, the foams were thermally decomposed in the NBS Toxicity Test Method apparatus via a two phase procedure previously shown to produce high concentrations of HCN. This report addresses the issue of whether the addition of a copper compound to a flexible polyurethane foam would affect the flammability characteristics of the foam. The following properties were examined: 1. ignitability in three systems [the NBS Toxicity Test Method, the Cone Calorimeter, and Lateral Ignition and Flame Spread Test (LIFT)], 2. heat release rate under small-scale (Cone Calorimeter) and medium-scale (furniture calorimeter), 3. smoke obscuration (Cone Calorimeter), and 4. rate of flame spread (LIFT). In all cases, no differences in flammability characteristics between the treated and untreated foam were observed.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

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